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## 825 Cytotoxicity of different root canal sealers and intracanal medicaments. An in-vitro study. B. BRISEÑO\*, B. WILLERSHAUSEN\* and D. SCHÄFER\*. Departments of Operative Dentistry\* and Otorhinolaryngology\*, Johannes Gutenberg University, Mainz, Germany

The biological effect and possible stimulation of inflammatory reactions of root canal sealers on surrounding tissues could be decisive for the outcome of a root canal treatment. The aim of this study was to determine the cytotoxicity of various ortho and retrograde root canal sealers as well as an intracanal medicament on cell growth as well as the production of inflammatory markers in vitro by means of cell cultures. Gingival fibroblasts between the 3th to 7th passage were cultured with different root canal sealers (AH Plus, RBA, Endion), a retrograde sealer (MTA) and a canal medicament (Ledemix). Wells with only fibroblasts served as negative controls. Apexil and gutta-percha points were used as positive controls. 15 wells (3x10<sup>5</sup> cells/well; 0.12 mg protein/ml) were covered and incubated with each sealer with a mean weight of 2.3 mg ( $\pm$  0.25 mg). Cell growth (protein mg/ml, cell number and vital staining) was determined after a period of 6 days of incubation. The inflammatory influence of the sealers was determined through the release of prostaglandin E<sub>2</sub> (PGE<sub>2</sub>, clone E2R1) with fibroblasts (15,000 cells/well) which were incubated during 30 min. In addition the fibroblasts cultures were stimulated with arachidonic acid (10<sup>-5</sup> M). The results were statistically analysed with the Kruskal-Wallis test ( $p=0.001$ ). The cell growth results showed significant differences between the tested materials and the positive and negative controls (cell staining, cell number). The release of lipid mediators as a sign of inflammation in comparison to the negative controls showed significant differences to most materials with exception of Apexil, MTA and gutta-percha. This results of this study provide information on possible inflammatory effects of root canal filling materials; thus, could be useful when selecting a root canal sealer with a lower irritation potential.

## 827 Effect of long-term water storage on the flexural properties of fiber-reinforced composite. P.K. VALLITTU\* (University of Turku, Institute of Dentistry & Biomaterials Project, Turku, Finland).

Previously, it has been reported the effect of water storage for 48 weeks on the flexural properties of fiber-reinforced composite (FRC) used in dentistry. It was suggested that the reduction in flexural strength of E-glass FRC was mainly caused by the plasticization of the polymer matrix of the FRC rather than corrosion of the surface of the glass. The aim of this study was to evaluate the flexural properties of FRC materials after water storage of 180 weeks. Continuous woven silanized E-glass and woven silica fibers were used to reinforce heat-cured and autopolymerized denture base polymers. Fibers were oriented at a 45-degree angle to the long axis of the test specimens. Dry specimens and those stored in distilled water at 37°C for up to 180 weeks were tested with a three-point loading apparatus. The flexural properties (strength, MPa / modulus, GPa) were:

MATERIAL	DRY	WET (after 180 wks)	p (ANOVA, n=5)
Heat-cured	98 / 2.9	76 / 2.7	<0.001 / 0.007
Heat-cured+E-glass	195 / 9.4	142 / 8.5	0.004 / 0.413
Heat-cured+silica	188 / 8.5	117 / 7.9	<0.001 / 0.024
Autopol.	89 / 2.9	64 / 2.5	<0.001 / <0.001
Autopol.+E-glass	299 / 10.8	215 / 9.9	<0.001 / 0.394
Autopol.+silica	289 / 10.3	166 / 8.2	<0.001 / 0.003

Most of the reduction in flexural properties of all the materials occurred during the one month water storage. Post-hoc analysis revealed that after the storage of one month, no statistically significant reduction was found. This was assumed to show the reversible plasticization effect of water. The results of this study suggest that flexural strength of FRC is reduced during one month storage in water and remains approximately at that level for 180 weeks.

## 829 Water Uptake Behaviour of a Heterocyclic Methacrylate System from Solutions. R. SHAH\*, M.P. PATEL, M. BRADEN. (IRC in Biomedical Materials, QMW, London, UK).

Earlier studies have shown that self-cured poly(ethyl methacrylate)/tetrahydrofurfuryl methacrylate (PEM/THFM), possesses unique water absorption characteristics. Its water uptake behaviour from various biological solutions has been studied by Sawtell *et al.* (J. Mater. Sci. Mater. Med. 8, 1997). Hence, the current work investigates the detailed effect on uptake of varying concentrations of glucose and sodium chloride (NaCl) as external solutions. Rectangular samples (40x10x1mm<sup>3</sup>, duplicate) of the polymer were prepared, by mixing 5g of PEM with 3 ml of THFM, and immersed in 100ml of each solution (at 37°C). Uptake was measured gravimetrically. The equilibrium uptake decreased from ~30% in water to ~2% at an osmolality of ~60mM. Correspondingly, the diffusion coefficient increased (see Table). This critical dependence of uptake on the osmolality of the external solution indicates that the polymer contains osmotically active sites, confirming earlier studies. The water uptake of PEM/THFM is critically dependent on the osmolality of the external solution up to ~40mM.

solution/ concentration (mM)		percentage water uptake		diffusion coefficient (m <sup>2</sup> /s)	
NaCl*	Glucose	NaCl*	Glucose	NaCl*	Glucose
30	60	1.96	2.25	$3.31 \times 10^{-11}$	$3.44 \times 10^{-11}$
40	80	2.02	1.99	$3.66 \times 10^{-11}$	$3.88 \times 10^{-11}$
50	100	1.95	1.86	$3.60 \times 10^{-11}$	$3.94 \times 10^{-11}$
60	120	2.05	1.72	$4.43 \times 10^{-11}$	$3.96 \times 10^{-11}$

Table: Water uptake from solutions (\*dissociation constant of NaCl = 1 so concentration is halved). The support of the EPSRC, UK, to the IRC in Biomedical Materials is gratefully acknowledged.

## 831 Water Uptake of Polymeric Drug Release Systems. R. ARSLANOGLU\*, A. CLOUGH, M. BRADEN, M.P. PATEL. (University of Surrey, Guildford, UK, Queen Mary and Westfield College, London, UK).

Room temperature polymerising/cross-linking polymers have great potential as drug delivery systems for the treatment of oral diseases such as candidal infections (Wilson and Wilson, J. Oral Rehab., 20, 1993). However, drug release is contingent on water absorption by the polymer. Three different non-degradable polymer/drug systems were therefore studied with respect to water uptake, namely, i) room temperature polymerising poly(ethyl methacrylate)-tetrahydrofurfuryl methacrylate (PEM/THFM), ii) a condensation cured silicone and iii) an addition cured silicone. Chlorhexidine diacetate (CHD) was added to each system by simply blending at 9% w/w, and samples were prepared in triplicate for both drugged and control systems. Water uptake was measured gravimetrically over a period of 6 weeks. In each case, the presence of CHD enhanced the water uptake. The water uptake of the PEM/THFM system with 9% CHD was linear with respect to  $t^{1/2}$ , attaining an uptake of ~10% whilst the control group reached ~1.7% in the first 8 hours and remained constant. The water uptake of the condensation cured silicone system was similar to the control and drugged system up to 24 hours, at ~3%, but subsequently the drug containing system accelerated rapidly over 30%, which was 5 times greater than the corresponding control system. However, in the case of the addition cured silicone, equilibrium was achieved within 24 hours at ~1% for the control, whereas with 9% CHD the system initially lost weight for the first 20 minutes, indicating the loss soluble material, but subsequently attained an uptake of 2.5%. The difference in uptake between the two silicone materials may be related to alcohol formation in the condensation silicone. The presence of CHD generally increased the water uptake of the three systems studied which was linear with respect to  $t^{1/2}$ , indicating diffusion control. There were marked differences between the two silicone systems studied. The support of the EPSRC, UK is gratefully acknowledged.

## 826 Aspects of Water Sorption in Filled Resin Restorative Materials. L. Musanje\*, B.W. Darvell (Faculty of Dentistry, The University of Hong Kong).

Water uptake is known to have marked effects on the physical and mechanical properties of filled resin restorative materials (FRRMs). For test results to be interpretable, conditions need to be standardized and with reference to normal oral working conditions. Two types of visible light cured FRRM, Heliomolar radiopaque (HR) and Tetric Ceram (TC) (Ivoclar, Schaan, Liechtenstein) were studied. Specimens (20 x 1.5 x 1.0 mm<sup>3</sup>) were cured (Luxor-4000, Imperial Chemical Industries, Macclesfield, Cheshire, UK) at 5 overlapping locations for 60 s per spot, randomly distributed into 3 groups, 6 specimens each. The 3 groups were separately exposed to water vapour (WV), deionized water (DW) and artificial saliva (AS) (V.W.H. Leung and B.W. Darvell; J Chem Soc Faraday Trans 87(11): 1759 - 1764, 1991) and stored at 37°C. Specimens exposed to WV were later immersed in DW, after gaining equilibrium mass. Fluids were replaced every 5 d. A rapid gain in mass, followed by a gradual loss, was observed as a general pattern for both DW and AS for both materials. The average time to peak mass in DW and AS was 7 d for both materials. The peak gain in mass by HR exposed to DW and AS was 0.95% and 0.90%, for TC 0.51% and 0.50%, respectively. Specimens exposed to WV equilibrated in 25 d; the gain in mass was 0.76% for HR and 0.47% for TC. These peak masses remained stable while exposed to WV, but when exposed to DW a small gain was followed by a gradual but continuous loss in mass. 1. For specimens originally exposed to WV, the loss in mass that occurred after exposure to DW can only be attributed to loss of solubles from FRRMs. 2. Loss of solubles is a continuous process that must start immediately on exposure to water or to artificial saliva. 3. The loss of mass was greater in AS than in DW after long term exposure suggesting hydrolysis. Supported by HKU Grad. Sch. Res. Stud Support Fund.

## 828 Water uptake of some methacrylate homopolymers. S. PARKER\*, P.D. RIGGS and M. BRADEN (IRC in Biomedical Materials, St. Bartholomew's & the Royal London School of Medicine & Dentistry, QMW, London, UK)

Methacrylate monomers and polymers are the main components of many materials used in dentistry, from denture base to composite filling materials to soft lining materials. The aim of this study was to evaluate the water uptake of some novel soft lining materials (Arist-1054 & 1055 IADR 1998), n-hexyl (nHMA), ethyl hexyl (EHMA), 1-tridecyl (TDMA) and C17.4 (C17.4MA) methacrylate, ethylene glycol (EGDMA), 1,6 hexanediol (HDMA) and 1,12 dodecanediol (DDMA) dimethacrylate. nHMA, EHMA, TDMA and EGDMA were supplied by Bonar Polymers Ltd. C17.4MA, HDMA and DDMA were supplied by Huls Chemicals. 1% lauryl peroxide was added to each monomer and specimens 20mm x 40mm x 1mm were produced by the method used by Patel *et al.* Biomaterials, 8, 53 1987. Two specimens of each were used and immersed in distilled water at 37°C, weight change with time was recorded. After 6 months specimens were desorbed in an oven at 37°C and solubility calculated.

Material	nHMA	EHMA	TDMA	C17.4MA	EGDMA	HDMA	DDMA
% wt change	0.20	-0.17	-0.50	-0.11	3.08	0.39	0.63
% solubility	0.02	0.68	0.92	0.44	1.73	0.58	0.21
%Total uptake	0.22	0.51	0.42	0.33	4.82	0.97	0.84

Kalachandran and Kusy (Polymer, 32, 2428, 1991) noted that water uptake decreases with increasing length of side chain for the rigid methacrylates but not for elastomeric methacrylates, probably as a result of the presence of water soluble impurities. This study found that water uptake of the elastomeric methacrylates is low and did decrease with increase in length of side chain and water uptake of the dimethacrylates increased with decreasing distance between the methacrylate groups. The support by the EPSRC for the IRC in Biomedical Materials is gratefully acknowledged.

## 830 Modification of the Hydrophilicity of a Heterocyclic Methacrylate System. M.P. PATEL\*, M. JOHNSTONE, F.J. HUGHES and M. BRADEN. (IRC in Biomedical Materials, St. Barts. & the Royal London Sch. of Med. & Dent., QMW, London, UK).

Earlier studies have reported that a room temperature polymerising system based on poly(ethyl methacrylate)/tetrahydrofurfuryl methacrylate (PEM/THFM), was effective as a drug delivery system and a tissue repair material. The aim of this study was to investigate the effect of modifying the hydrophilicity of the system on water sorption, which will in turn enhance the drug release characteristics of the material. This was achieved by replacing 50 and 70% v/v of the THFM with hydroxyethyl methacrylate (HEMA), 50, 70 and 100% v/v with hydroxypropyl methacrylate (HPM), and the addition of chicken serum albumin (CSA, 0.625g/5g PEM), respectively. PEM (or PEM+CSA, 1g) was mixed with 0.6 ml of the appropriate monomer solution containing 2.5% N,N-dimethyl-p-toluidine v/v, and moulded into rectangular strips (~75x35x1mm, n=2). These were immersed in distilled water (37°C), removed and weighed at appropriate time intervals, to record the weight gained, until equilibrium was attained. Replacing THFM (in part) by HEMA or HPM modified the water uptake behaviour of PEM/THFM, with the uptakes at equilibrium (between 4 and 7.5%) being surprisingly less than that of the parent system itself (~30%). However, the diffusion coefficients increased by 2000-10000 fold (~10<sup>-11</sup> m<sup>2</sup>/sec for PEM/THFM, to 2-5.3x10<sup>-11</sup> for the HEMA systems and 3.76-9.62x10<sup>-11</sup> for the HPM systems), thus representing a change in the uptake mechanism from a cluster to a continuum dominated process. This may explain the enhanced release of recombinant human bone morphogenetic protein-2 when 10% THFM was replaced by HEMA, in an earlier study (IADR Abstr. #1061, 1999). CSA generally enhanced water uptake of all systems, as expected for a water-soluble additive, but the effect was greater with the less hydrophilic HPM. The addition of the hydrophilic monomers, HEMA and HPM, to the PEM/THFM system decreased water uptake and increased diffusion coefficients of the parent system. The addition of CSA increased water uptake but decreased the diffusion coefficients. The support of the EPSRC, UK, to the IRC in Biomedical Materials is gratefully acknowledged.

## 832 Absorption of experimental soft liners in water and saline. S.N. NAZHAT\*, S. PARKER and M. BRADEN. (Biomaterials in relation to dentistry, IRC in Biomedical Materials, QMW, University of London, E1 4NS, UK).

The absorption of filled and unfilled experimental soft liners was investigated in two environments, water and saline. An isoprene-styrene copolymer (Kraton D1111, Shell Chemicals) was mixed with methyl-methacrylate and hexanediol-dimethacrylate, HD (Huls Chemicals) at 50/50 wt/vol respectively with 1% lauryl peroxide as initiator. HD was added at 40 and 60% in the methacrylate mixture. Two similar compositions were filled at 5% with fumed silica, C687 (Central Chemicals) treated with  $\gamma$ -methylacryloxypropyl trimethoxysilane, MPS (A174, Union Carbide). HD reduces the uptake due to cross-linking and when combined with filling this property is enhanced (Trans. Soc. Biomaterials, 1998, 333). Absorption studies were carried out on specimens at 37°C in distilled water and 0.225M NaCl solution, and the change in weight was measured. Desorption studies were carried out at 37°C to calculate the solubility (Sol) and desorption diffusion coefficients ( $D_{des}$ ).

Carried out at 37 °C to calculate the solubility ( $S_{sol}$ ) and desorption diffusion coefficients ( $D_{des}$ )						
Material	Distilled Water after 279 days immersion			0.225M NaCl after 275 days immersion		
	Abs. (%wt)	Sol. (%wt)	$D_{des}$ (M <sup>2</sup> S <sup>-1</sup> )	Abs. (%wt)	Sol. (%wt)	$D_{des}$ (M <sup>2</sup> S <sup>-1</sup> )
40% HD	1.86	0.32	$3.89 \times 10^{-12}$	0.98	0.18	$6.09 \times 10^{-12}$
60% HD	0.76	0.27	$6.57 \times 10^{-12}$	0.61	0.15	$7.97 \times 10^{-12}$
40% HD+F	1.41	0.18	$5.18 \times 10^{-12}$	0.77	0.10	$7.88 \times 10^{-12}$
60% HD+F	0.91	0.23	$6.13 \times 10^{-12}$	0.79	0.15	$6.71 \times 10^{-12}$

All formulations gave low uptake materials in both environments (<2%).  $D_{des}$  were lower in saline due to the lower overall uptake, as with solubility values. This was due to its greater osmotic pressure. HD reduced the overall uptake, however, adding the filler at 60% HD increased the uptake in both environments, suggesting a threshold limit for effective filling and cross-linking. The support of the EPSRC UK of the IRC in Biomedical Materials is gratefully acknowledged.